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Athens, Alabama 35611

DRA

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National Aeronautics and Space Administration

George C. Marshall Space Flight Center

Marshall Space Flight Center, Alabama

Submitted herewith is the final report on Contract NAS8-28058 with Athens College. Chemical Analysis of outgassing contaminants on spacecraft surfaces. Principal investigator, Dr. Ronald C. McNutt.

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FINAL REPORT

Introduction:

Studies are continuing at Athens College to develop methods for analyzing and characterizing outgassing contaminants from such materials as RTV 501 potting compound and S 13 G paint. Similiar work is planned on other materials of interest to NASA that may contribute to contamination through outgassing in a low pressure environment. Work on RTV 501 materials is nearly complete except for obtaining high resolution spectra. More emphasis will be placed on S 13 G paint in the next contract period.

Fractional distillation of a gross distillate from RTV 501 rubber was carried out and the distilled fractions examined as to their ultraviolet and infrared spectra. In addition these fractions were examined by gas liquid chromatography. A sensitive technique for structural analysis and molecular identification, which should be useful in this effort, is a Gas Chromatography-Mass Spectroscopy system. We have investigated the possibility of obtaining such an instrument, but have found it economically unfeasible at this time.

Several sheets of S 13 G paint on aluminum were obtained from Space Sciences Laboratory. We have begun examination of this material.

The instruments obtained from Space Sciences Laboratory for high resolution spectroscopy, photochemical behavior and related

studies are now functional. We need only to complete the assembly of certain cell compartments to begin this phase of the work.

During the current contract period some oral consultations were held with certain Space Sciences Laboratory personnel concerning chemical aspects of their research. This type of assistance with chemical problems will be done on a more formal basis in the next contract period.

We had planned for one of our student assistants to give a presentation on the RTV 501 work at the Alabama Academy of Science annual meeting. There were, unfortunately, not enough chemical entries to warrant a chemistry section. The presentation will therefore not be given this year. We plan to obtain permission and report some of the work at the meeting next year.

Results and Discussion:

Samples of S 13 G paint on aluminum were obtained and extracted with cyclohexane. The cyclohexane was evaporated and the residual extract obtained. The extract was found to have general absorption in the ultraviolet spectrum (Fig. 1). This type of absorption is exhibited by extracts from most of the potting compounds and paints we have previously examined. The extract appeared to exhibit only one peak when a sample dissolved in acetone was put through a silicone column on the gas chromatograph (Fig. 2). The first peak is due to acetone. It was necessary to dilute the extract with solvent because it was too viscous to flow through the microliter syringe used in G.L.C. analysis. The infrared spectrum for the

S 13 G extract was obtained (Fig.3). This spectrum is very similiar to that for the base paint component of S 13 G paint and dissimiliar to the infrared spectrum for the S 13 G catalyst component. (See figures 5 and 6 for the final report of November 27, 1972.)

A more extensive investigation is planned for the S 13 G paint including, but not necessarily limited to, high resolution spectrographic techniques, photochemical behavior and mass spectral studies.

Work has been continuing on the distillation of volatile materials from RTV 501 rubber. These distillates are being examined and compared to extracts obtained by extraction of RTV 501 rubber with cyclohexane. Figures 4 and 5 show the infrared spectra for RTV 501 distillate and RTV 501 cyclohexane extract respectively. The spectra are very similiar. The gas chromatography results are somewhat more ambiguous. The extract shows only one chromatographic peak. This peak was at first thought to be the same as the first peak on a G.L.C. chromatogram of the RTV 501 gross distillate (Fig. 6) or from runs on fractional distillation cuts (Fig. 7). Close examination of the various G.L.C. graphs appear to indicate that the retention time for cyclohexane extract peak may be slightly less than the first peak from RTV 501 distillates. Further investigation of this problem is currently being pursued. The ultraviolet spectra for RTV 501 distillates and cyclohexane extracts are quite similiar. (See figures 5 and 6

of the May 26, 1972 final report.) We will consult with the Space Sciences Laboratory contract monitor for direction on this phase of the investigation. There are advantages for using extraction techniques such as simplicity of the method and the apparent purity of the extract; that is only one G.L.C. peak is observed. However, the distilling procedure under vacuum is more consistent with space conditions. We are hopeful that results to be obtained with the spectrographic equipment will be more definitive.

We have attempted molecular weight determinations on some of the distillates by freezing point depression techniques. A good solvent for this method has not been found; however, preliminary results in camphor and cyclohexane indicate molecular weights in the range from 110 to 175 a.m.u.

An interesting observation was made during the course of distilling volatile materials from the RTV 501 rubber. We found that if one extrapolates the temperature at which one first observes a distillate versus the pressure under which the vacuum system is operating there should be considerable volatile material outgassing at very low pressures in the temperature range of ordinary room conditions. However, the lowest pressure we have been able to attain with the present system was about 5×10^{-2} mm Hg. The RTV 501 rubber begins to outgas, by visual observation, at about 60° C at this pressure. We will make an effort to attain pressures that will allow us to obtain outgassing materials without heating to avoid decomposition of the distillates.

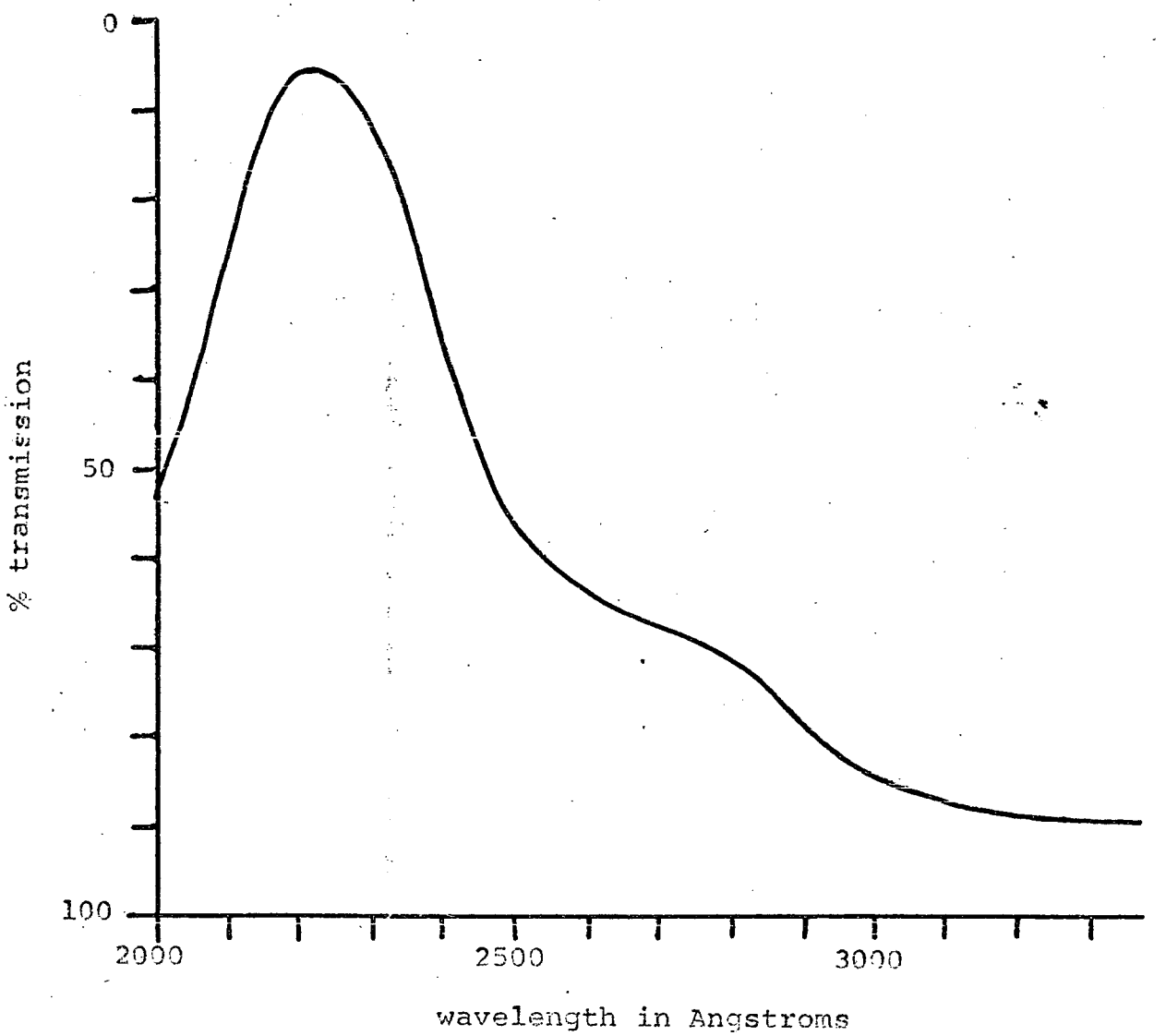
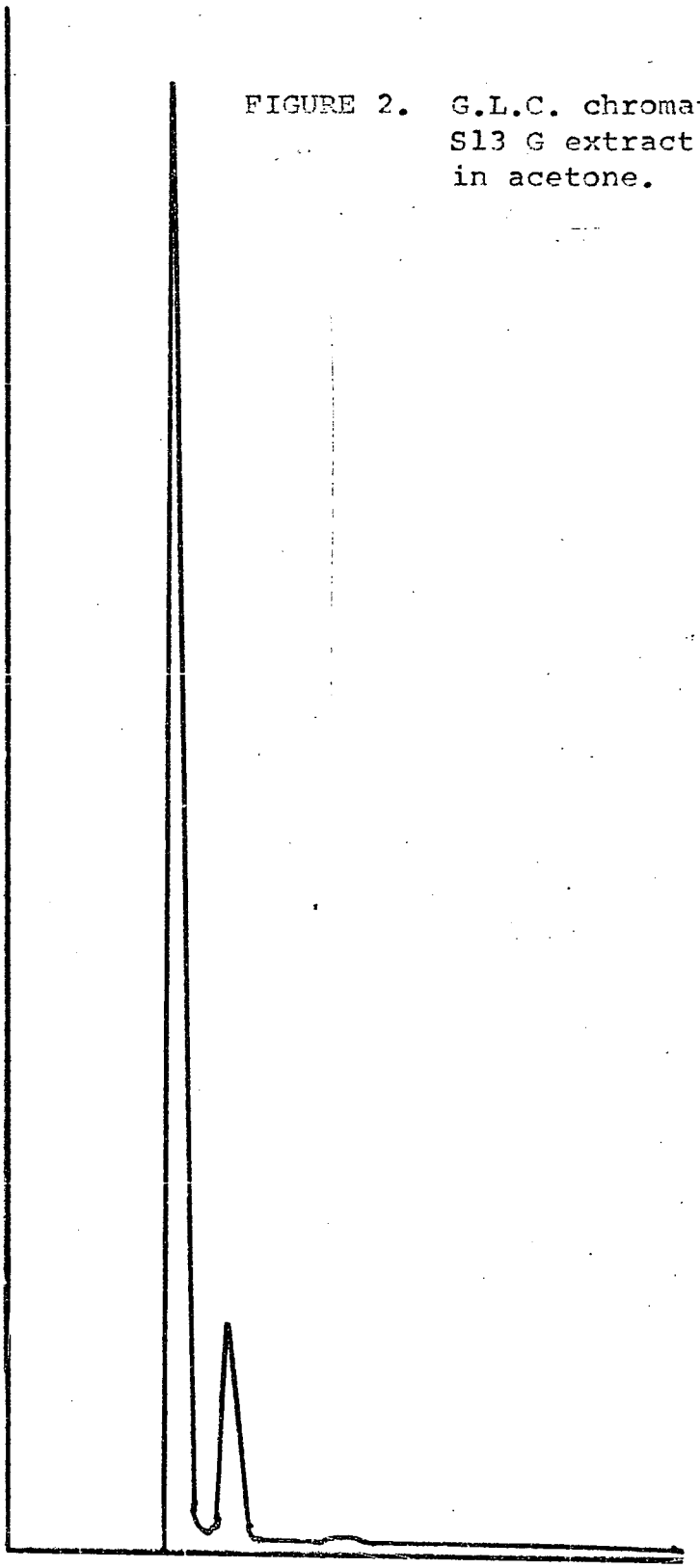


FIGURE 1. Ultraviolet spectrum for S13 G extract in cyclohexane.



retention time

FIGURE 2. G.L.C. chromatogram of S13 G extract dissolved in acetone.

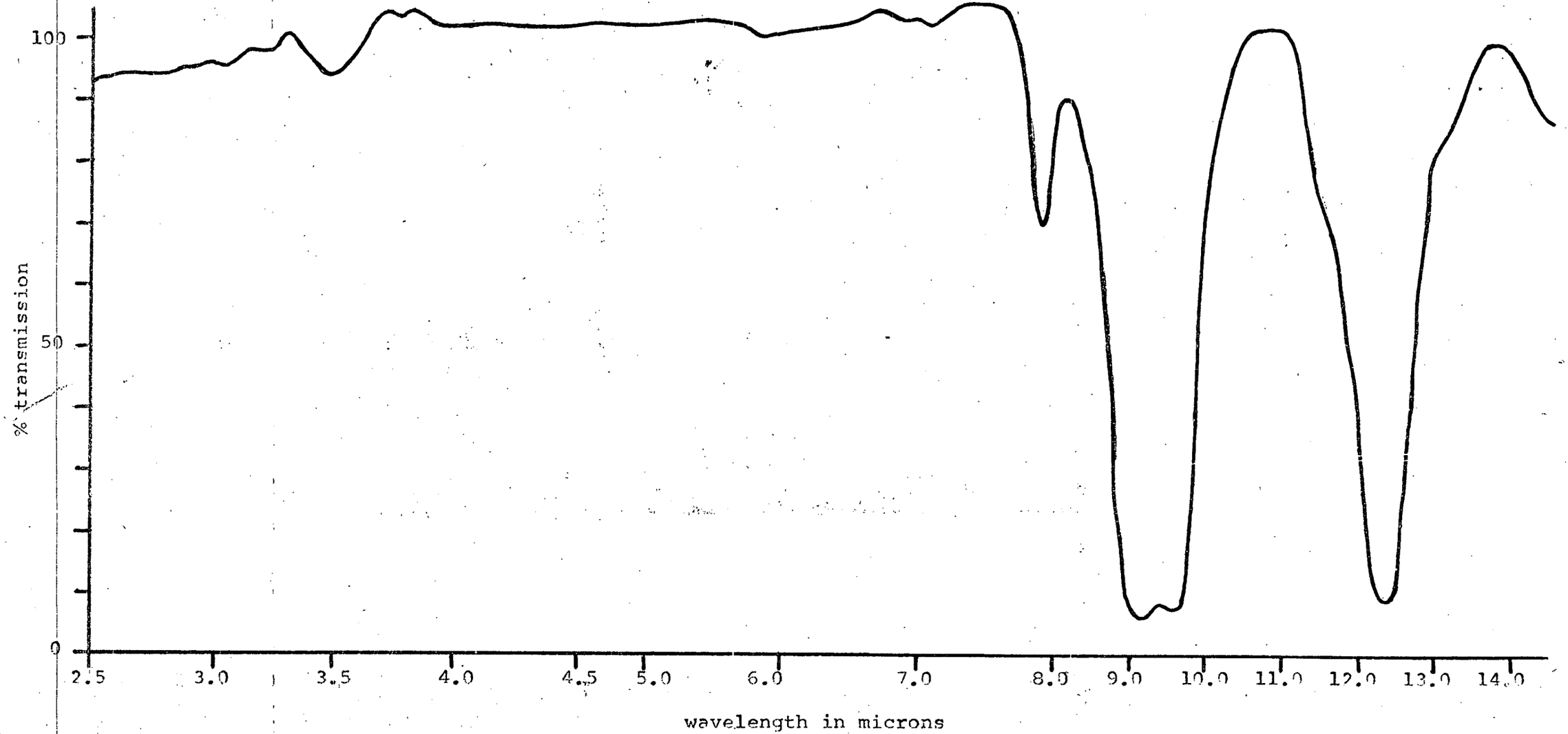


FIGURE 3. Infrared spectrum for S13 G extract in cyclohexane. Cyclohexane in reference cell (1mm).

FOLDOUT FRAME 1

FOLDOUT FRAME 2

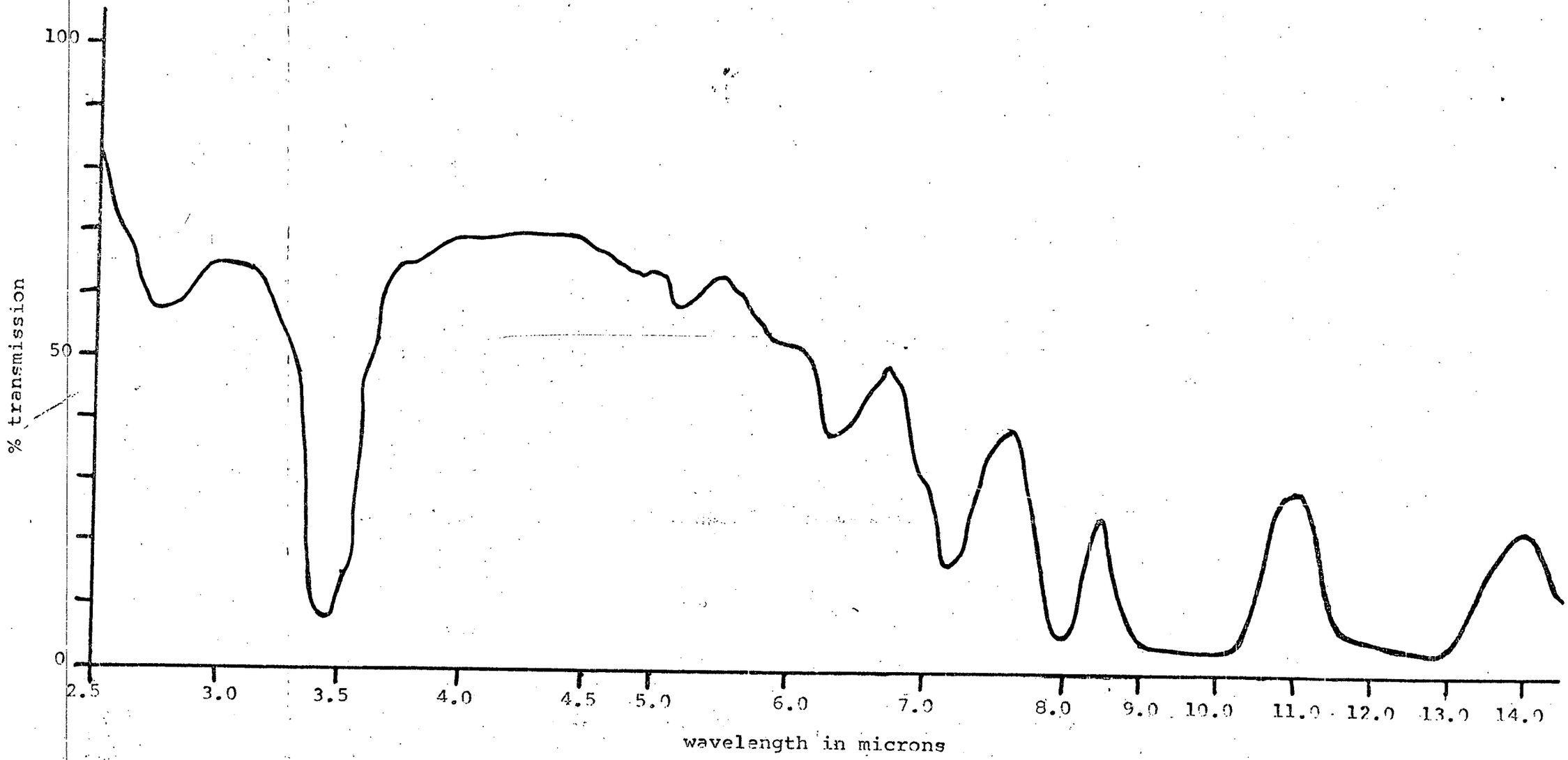


FIGURE 4. Infrared spectrum for cyclohexane extract of RTV 501 on NaCl cell.

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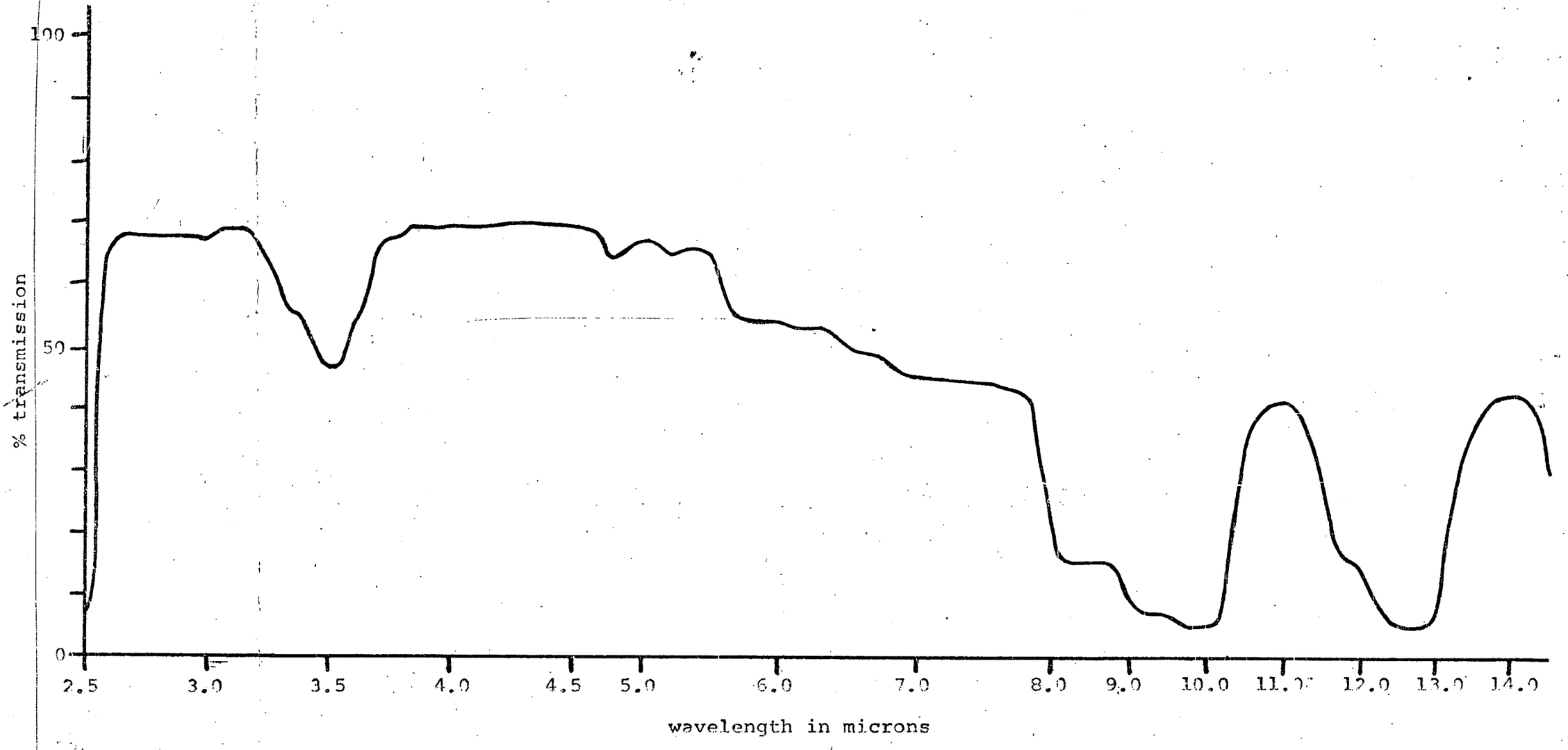

FIGURE 5. Infrared spectrum for RTV 501 distillate on NaCl cell.

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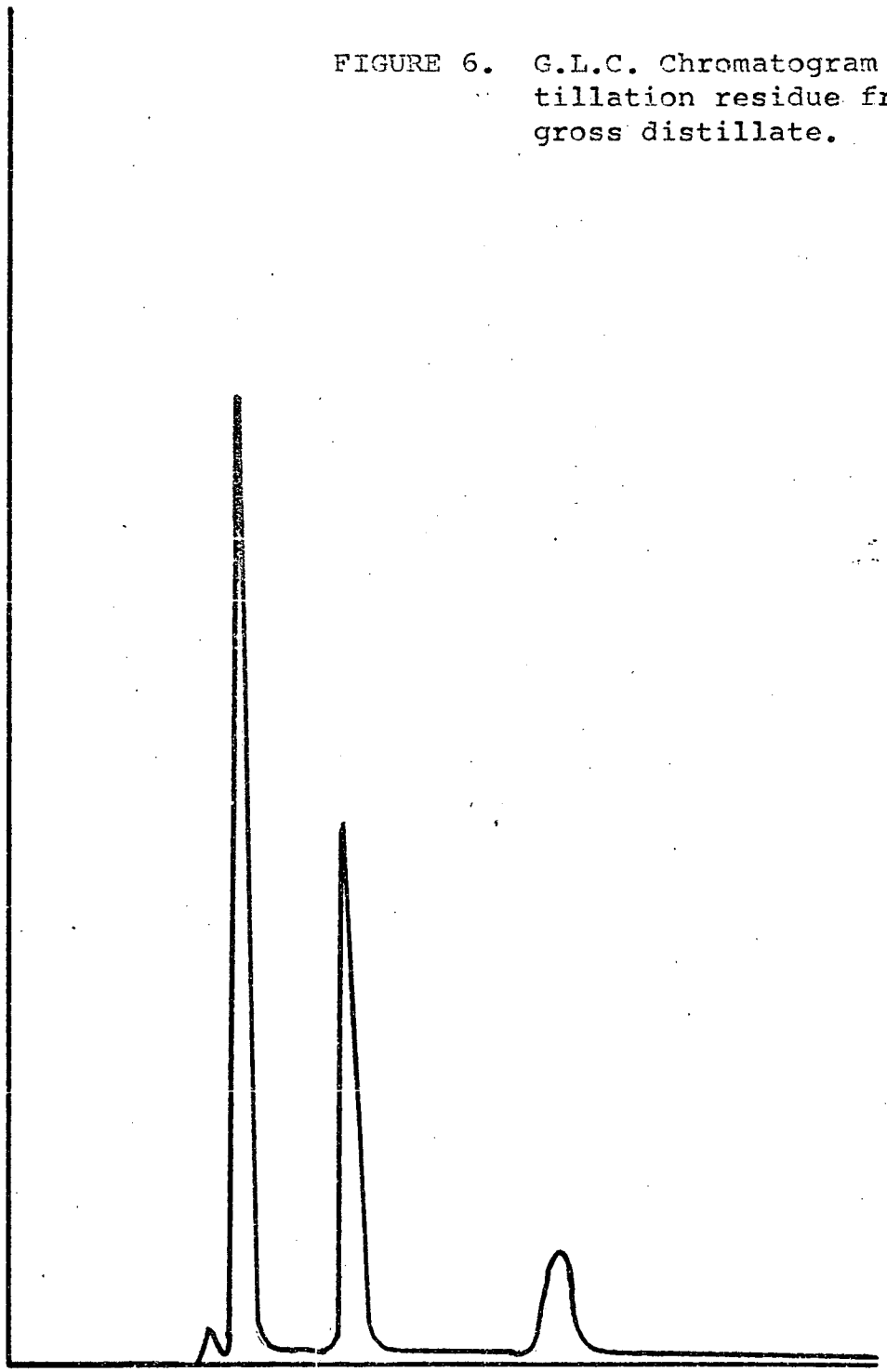
FIGURE 6. G.L.C. Chromatogram of distillation residue from RTV 501 gross distillate.



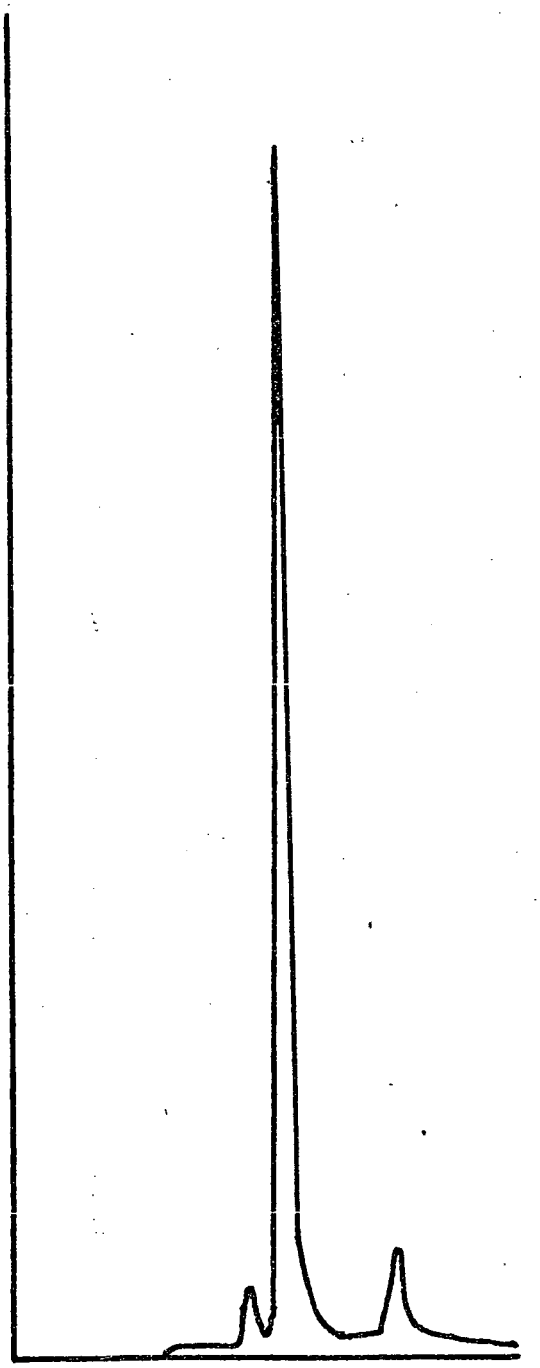


Figure 7. G.L.C. Chromatogram of a fractional distillation cut from RTV 501 gross distillate.